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(57) In a combined scanning near field optical microscope (Nsom) and atomic force microscope (AFM), an optical fibre probe (1) which has a minute opening on the top of a sharpened tip is brought close to a sample (2), and the probe is moved by a piezo actuator (15) along x- and y-axis directions so that a minute spot beam emanating from the minute opening can scan over the sample. For circular polarisation modulation to be incorporated in the process, a beam is given an optical delay, before it is incident on the optical fibre probe (1),

changing at a frequency of  $p$  (Hz) by means of a piezo-optical modulator (10). A minute spot beam emanating from the minute opening passes through the sample (2) to be received after passage through the sample (2) to be received after passage through an analyser (5) by a light receiving element (7). The output from the light receiving element (7) is fed to a lock-in amplifier,  $p$ - and  $2p$ -components are separated through lock-in rectification, and they are rendered into images by a controller (16). It is used for measuring the distribution of magneto-optical effects.

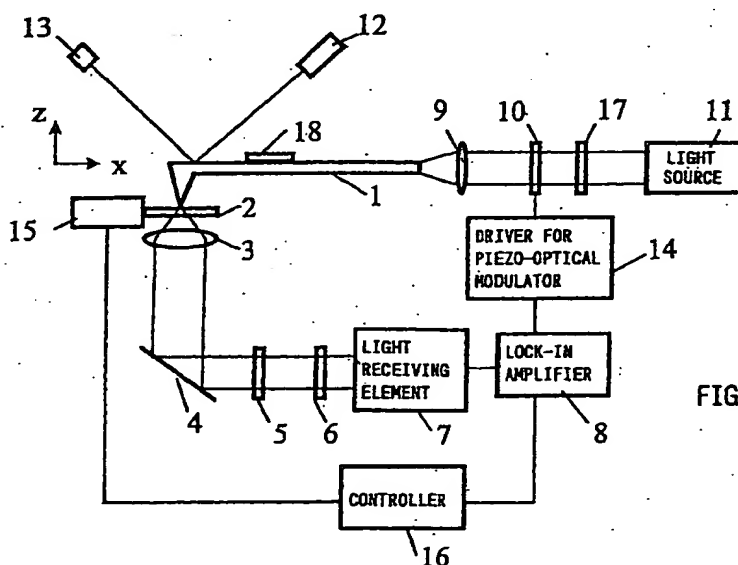


FIG. 1

## Description

This invention relates to a measuring apparatus which can measure the polarising activity and the distribution thereof of a test substance with a high resolution, by detecting a beam which has interacted with a tiny area of a substance at the tip of a probe, and by utilising the polarisation characteristics which the beam presents.

It becomes important to observe, for various test samples, the distribution of their optical activities (circular dichroism and optical rotation) in very tiny areas, and to obtain quantitative evaluations of those optical activities. Such optical activities include natural optical activities, electro-optical activities, magneto-optical activities, and piezo-optical activities, and with the recent technical advance in the field of memories having a gigantic capacity such as a hard disk, opto-magnetic disk, etc., a demand for equipment allowing a precise observation and measurement of magneto-optical effects has rapidly become intense.

For example, to observe the distribution of magneto-optic effects as one aspect of such optical activities with a high precision requires the observation of magnetic sectors and barriers. The well-known method used for this purpose includes polarised light microscopy, Lorenz transmission electron microscopy, spin-polarised scanning electron microscopy, and magnetic force microscopy. A recent article reports an observation of the magnetic barriers of a vertically magnetised membrane by the use of a scanning near field optical microscope (APPLIED OPTICS, Vol. 31, No. 22, 1992, p. 4563, E. Betzig et al.).

Here a scanning near field optical microscope will be briefly described. A widely available method consists of sharpening an optic fibre or a beam transmitting body, and preparing a minute opening at its tip having a diameter equal to or less than the wavelength of the beam. By the same method with which a conventional scanning atomic force microscope or a scanning tunnel microscope adjusts the distance between a cantilever and a sample, the minute opening is placed so close to the surface of a sample that the distance there between is equal to or less than the wavelength of the beam. By introducing, while maintaining the above state, a beam into the optic fibre with such a minute opening, radiating a tiny area of a sample with the beam emanating from the minute opening, and scanning the beam over the sample in a two-dimensional plane, a microscope can achieve a high resolution microscopy in accordance with the size of the minute opening. In the example mentioned earlier where a scanning near field optical microscope was used for the observation of magnetic barriers, a linearly polarised beam emanating from a minute opening is allowed to radiate a sample, and the beam transmitting through the sample is received by an analyser (cross-Nichol method).

On the other hand, a method to quantitatively determine the circular dichroism or optical rotation of a sample, for example, on the basis of magneto-optical effects (methods dependent on other optical activities work on essentially the same principle) is described in detail in "Light and magnetism" published by Asakura Publishing Co. (written by Sato, K.). The optical rotation due to magnetism can be determined by perpendicularly intersecting polarisers (cross-Nichol method), Faraday-cell method, and a rotational analyser. The use of a quarter-wave plate will allow the measurement of the circular dichroism of the sample. Further, modulation of a circularly polarised beam (circular polarisation modulation) will enable the measurement of both the magneto-optical rotation and magneto-optical circular dichroism with a high sensitivity.

Here, circular polarisation modulation will be briefly described with reference to Fig. 2. A linearly polarised beam having passed through a linear polariser 101 is given, by a piezo-optical modulator 102 working on birefringence, an optical delay which changes at a frequency of  $p$  (Hz). Then, the same beam, after having been reflected from or passed through a sample 103 (the beam is reflected from the sample in Fig. 2), is allowed to pass through an analyser 104 to reach a light receiving element 105 for registration. From the  $p$  (Hz) component and  $2p$  (Hz) component of the beam having passed through the analyser 104, it is possible to determine the circular dichroism and optical rotation the beam has undergone, respectively.

The principle underlying circular polarisation modulation will be described by equations. For brevity, the direction along which a beam travels is assumed to coincide with the  $z$ -axis. Assume that in Fig. 2 the linear polariser 101 has an angle of  $45^\circ$  with respect to  $x$ -axis. The electric field  $E_1$  of the beam having passed through the linear polariser 101 can be expressed by:

$$E_1 \propto (i + j) \quad (1)$$

given that  $i$  and  $j$  are the unit vectors of  $x$ - and  $y$ -axes respectively.

Given that there is a delay of  $\delta$  between  $x$ - and  $y$ -components of the electric field  $E_2$  of the beam which has passed through the piezo-optical modulator 102,

$$E_2 \propto \{i + \exp(i\delta)\} \quad (2)$$

Assuming that the unit vectors of right- and left-circularly polarised beams are expressed by the following equations respectively:

$$r = (i + j)/2^{1/2}$$

$$l = (i - j)/2^{1/2}$$

then,  $E_2$  can be expressed by the following equation:

$$E_2 \propto \{1 - \exp(i\delta)\} r + (1 + \exp(i\delta)) l \quad (3)$$

Suppose that the complexly expressed amplitude reflections of right- and left-circularly polarised beams are expressed by  $r + \exp(i\theta_+)$  and  $r - \exp(i\theta_-)$  respectively, then the electric field  $E_3$  of reflected beam can be expressed by:

$$E_3 \propto \{(1 - \exp(i\delta)) r + \exp(i\theta_+) r + (1 + \exp(i\delta)) r - \exp(i\theta_-) l\} \quad (4)$$

The intensity  $I$  of the beam emanating from the analyser having an angle of  $\phi$  with respect to the x-axis is expressed by:

$$I \propto \{R + (\Delta R/2) \sin \delta + R \sin(\Delta\theta + 2\phi) \cos \delta\} \quad (5)$$

where

$$R = (r+2 + r-2)/2$$

$$\Delta R = r+2 - r-2$$

$$\Delta\theta = \theta_+ - \theta_- = -2\theta_k$$

$$\Delta R/R = 4\eta_k$$

and where  $\theta_k$  represents a Kerr's rotation angle and  $\eta_k$  a Kerr's ellipticity. Assuming that  $\phi = 0$ , and  $\Delta\theta$  is sufficiently small,  $\delta \sim \sin^2 \pi p t$ . Then, the equation can be resolved by the use of Bessel function into:

$$I \sim I(0) + I(p) \sin 2\pi p t + I(2p) \cos 4\pi p t + \quad (6)$$

In this equation,  $I(0)$ ,  $I(p)$ , and  $I(2p)$  represent factors respectively containing 0th- order, 1st-order and 2nd order Bessel functions, and

$$I(p) \propto \eta_k, I(2p) \propto \theta_k \quad (7)$$

Therefore, the  $p$ (Hz) component gives the Kerr's ellipticity and the  $2p$  (Hz) gives the Kerr's rotation angle. For details, see the above-described "Light and magnetism."

The above-described various methods employed for the observation of minute magnetic sectors have a number of problems as will be described. For example, polarised light microscopy, operating in the same manner as conventional optical microscopy, has its resolution restricted by the diffraction limit of the beam used, and only achieves a resolution that allows detection to a width of about half the wavelength of the beam used. Further, as it depends on the cross-Nichols method for detecting the optical activities of a sample, its detection sensitivity is low. Lorenz transmission electron microscopy has a resolution sufficiently high to distinguish about 10nm intervals, but it is only applicable to a thinly sectioned sample. Spin-polarised scanning electron microscopy has a problem in that it requires a large cost for instalment. Magnetic force microscopy has a considerably high resolution that allows discrimination of several tens of nm intervals, but it can scarcely be applied for the quantitative determination of the magnitude of a magnetic field or magnetisation. The scanning near field optical microscope has its resolution determined principally by the diameter of the opening of the probe, and has a considerably high resolution. The conventional minute spot scanning microscopy, however, usually depends for the detection of optical activities of a sample, on the cross Nichols method, and presents the following problems. It allows only a low sensitivity. Notwithstanding that the closer the minute spot beam emanating from a minute opening is to a linearly polarised beam, the higher the detection sensitivity, the minute spot beam emanating from a minute opening is usually elliptically polarised. This may form another cause for lowered sensitivity.

Among the apparatuses for quantifying various magneto-optical effects, there are some that allow the very sensitive quantification of a rotation angle through modulation, for example, by the use of a rotating analyser. This method, however, can not be applied to a tiny area exceeding the typical level handled by a conventional optical microscope.

As illustrated above by referring to the microscopic observation of magneto-optical effects as an example, the conventional methods whereby the distribution and quantification of optical activities of a sample have been obtained have more or less defects to be corrected, although some are advantageous in sensitivity, resolution and tolerance of sample handling, and others are advantageous in cost. What is mentioned above applies to the measurement not only of magneto-optical effects but also of optical activities at large. In view of this the object of the present invention is to provide an apparatus with which it is possible to observe/measure the optical activities of a sample with a high resolution and sensitivity, at a low cost, quantitatively, and without imposing any restrictions on the handling of the sample.

It is an object of the present invention to provide a scanning near field optical microscope based on polarised light which requires only a low cost for production because of its being based on the constitution of scanning atomic force microscopy, and allows observation of a sample without imposing any restrictions on the preparation thereof.

It is another object of the present invention to provide a scanning near field optical microscope where the size of a beam to radiate a sample or emanating from a sample is determined by the size of tip according to the principle underlying the scanning near field optical microscope. Thus it allows the observation of a sample with a very high resolution without being affected by a diffraction limit.

It is another object of the present invention to provide a scanning near field optical microscope which can achieve a highly sensitive circularly polarised modulation by giving a periodically changing optical delay to a beam.

It is a further object of the present invention to provide a scanning near field optical microscope where it is possible to reduce the changes in polarisation state due to external disturbing sources when the probe consists of a light transmitting body made of a material having a smaller photo-elasticity coefficient, and it can minimise the adverse effects due to stresses developed as a result of bending when the same material is applied for the preparation of a probe with a hook-like shape.

According to a first aspect of the present invention there is provided a scanning near field optical microscope comprising: a light source; a probe having a tip; means operatively to maintain the interval between the tip and a sample surface or medium within an action distance within which an interactive force results between the tip and the surface; means to obtain a polarised beam carrying optical information concerning the sample surface or medium, by allowing a beam from the light source to interact with the surface adjacent the tip; light receiving means to receive the polarised beam; modulating means placed in the light path between the light source and the light receiving means to establish a periodically changing optical delay in the beam.

Preferably the probe is allowed to have a light transmitting body.

Preferably the means which maintains the tip so close to the surface of a sample that the interval therebetween is kept within an action distance which allows and inter-atomic interaction or interactions by way of other elements further comprises:

a moving means which alters the distance between the tip and the surface;  
a distance determining means which determines the distance between the tip and the surface; and  
a control means which maintains the distance between the tip and the surface constant based on a signal delivered by the distance determining means.

Preferably the distance determining means still further comprises:

a vibrating means which vibrates the tip and the surface relative to each other in a horizontal or vertical direction; and a displacement detecting means which detects the displacement of the tip.

Preferably the modulating means which is inserted on a light path between the source and the light receiving means, and gives an optical delay changing at a regular cycle acts as a means to generate a periodically changing stress in the light transmitting body.

Preferably the present invention further comprises:

a light source;

a probe having a sharpened tip and a light transmitting body;

a means which maintains the tip so close to the surface of a sample that the interval therebetween is kept within an action distance which allows an inter-atomic interaction or interactions by way of other elements to be present between the tip and the surface;

a means which obtains a polarised beam carrying the data of optical activities of a tiny area of the sample or of a medium, by allowing a beam emanating from the source to interact with the sample surface at the tip; and a light receiving means which receives the polarised beam, wherein;

the material constituting the light transmitting body has a photo-elasticity coefficient of  $10 \times 10^{-6} [\text{mm}^2 \cdot \text{N}^{-1}]$  or less.

Preferably the material constituting the light transmitting body includes quartz glass containing lead oxide.

Embodiments of the present invention will now be described by way of further example only and with reference to the accompanying drawings, in which:

Fig. 1 is an explanatory view showing the constitution of Example 1 of a scanning near field optical microscope based on polarised light according to this invention.

Fig. 2 is an explanatory view showing the constitution of element necessary for a conventional method for measuring magneto-optical effects based on circular polarisation modulation.

Fig. 3 is an explanatory view showing the constitution of Example 2 of a scanning near field optical microscope based on polarised light according to this invention.

Fig. 4 is an explanatory view showing the constitution of Example 3 of a scanning near field optical microscope based on polarised light according to this invention.

Fig. 5 is an explanatory view showing the constitution of Example 4 of a scanning near field optical microscope based on polarised light according to this invention.

#### (1) Example 1

Fig. 1 illustrates the constitution of Example 1 of this invention. The basic constitution is the same as that of a conventional scanning near field optical microscope. The figure shows an apparatus which incorporates an optic fibre probe which is produced after an optic fibre acting as a light transmitting body has a minute opening prepared at its tip, wherein a minute spot beam emanating from the minute opening radiates a sample (illumination mode) and the beam after passing through the sample is registered (transmission type). Description will be given with reference to this example.

Firstly, a conventional minute spot scanning microscopy will be described with regard to its constitution and operation. A light source 11, comprising a gas laser, solid compound-based laser, or semiconductor-based laser, generates a light flux which passes through a polarisation adjusting element 17 consisting of a wavelength plate, and a piezo-optical modulator 10, to be incident, through the intervention of a fibre coupler 9, on the input end of a fibre optic probe 1. The optic fibre probe 1 is usually made of a single mode fibre, and its other end has its tip sharpened and circumference coated by a film composed of a metal like gold or aluminum, such that the tip has a minute opening whose diameter is equal to or less than the wavelength of the beam. The beam incident on the input end of optical fibre probe 1 emanates from the minute opening as a minute spot beam. The optic fibre may be made of a multi-mode fibre or a single hollow fibre, instead of a single mode fibre. Further, the optical fibre probe 1 has a part close to the tip bent like a letter L, and is attached on the surface of a piezo-electric element such as a bimorph or a quartz vibrator. It is possible to operate the apparatus in an AFM mode, or a dynamic mode often used in conjunction with scanning atomic force microscopy (AFM), by vibrating the optical fibre probe 1 vertically with respect to a sample by means of the piezo-electric element 18.

A sample 2 is placed on a piezo actuator 15 which can move along x-, y- and z-axis directions, and a controller 55 controls the movement of the piezo actuator 15. The controller, while maintaining constant the distance between the sample 2 and the tip of the optical fibre probe 1, scans the beam over the sample 2 in x- and y-axis directions using the piezo actuator 15. In this example, a method based on a device generally called an optical lever is used for determining the distance between the sample 2 and the tip of the optical fibre probe 1. This method consists of converging

a beam emanating from a laser source 12 onto the surface of a mirror placed close to the tip of the optical fibre probe 1, of receiving the reflected beam with a bisected light receiving element 13, and of determining the difference between the intensities of beams received by respective bisected segments, thereby to monitor the vibration state (frequency, amplitude and phase of the vibration) of the optical fibre probe 1. For example, when the optical fibre probe 1 comes close to the sample 2, its vibration state undergoes a change in the presence of forces resulting from inter-atomic interactions. Therefore, by adjusting the movement of the piezo actuator 15 along the z-axis direction in such a way as to allow the optical fibre probe 1 to make a vibration with a constant amplitude, it is possible to maintain constant the distance between the surface of sample 2 and the tip of the optical fibre probe. Thus, while maintaining constant the distance between the sample 2 and the tip of the optical fibre probe 1, it is possible, by scanning the beam over the sample, by moving the piezo actuator 15 in x- and y-axis directions and by monitoring how much the piezo actuator 15 moves along the z-axis direction, to obtain an image of the surface texture of the sample 2.

As the tip of the optical fibre probe 1 is positioned close to the surface of sample 2, the minute spot beam emanating from the minute opening transmits through the sample 2, is converged by a converging lens 3, has its path bent by a mirror 4, and passes through an analyser 5 and filter 6 to be received by a light receiving element 7. The filter 6 placed in front of the light receiving element 7 is to cut off the laser beam 12 which acts as one arm of the optical lever.

As the light source 11 usually consists of a laser source based on a gas or solid molecule, it often happens that a linearly polarised beam impinges on the optical fibre probe 1. But, the optical fibre probe 1 generally contains elements which may resolve the polarisation state or retard the phase, of an incident beam, and thus the minute spot beam emanating from the minute opening often suffers a degraded polarisation or becomes an elliptically polarised beam, notwithstanding that the incident light is a linearly polarised beam. When such a polarised beam is radiated upon the sample 2, and its polarisation state is monitored by a cross-Nichols method, the overall sensitivity will become low. To avoid such inconvenience, it is necessary to insert a wavelength plate or compensation plate, that is, an agent to cause an appropriate retardation, on the incident path of the optical fibre probe 1, thereby to adjust the polarisation state of incident light. Through this procedure it is possible to obtain a minute spot beam with a practically linear polarisation.

In spite of above fact, this invention adopts circular polarisation modulation dependent on a cross-Nichols method which is principally very sensitive. The method adopted in this example whereby an optical delay is given to a minute spot beam emanating from the minute opening of optical fibre probe 1 in accordance with a modulation frequency  $p$  (Hz) depends on the use of a piezo-optical modulator (PEM) 10 working on birefringence which incorporates an optically active crystal such as quartz or the like. A drive 14 to drive the PEM not only activates the piezo-optical modulator 10 but delivers a reference signal with respect to which a lock-in amplifier 8 performs a lock-in rectification. A light flux emanating from the light source 11 passes through the piezo-optical modulator 10 to be given an optical delay there, and is incident through the intervention of fibre coupler 9 on the input end of the optical fibre probe 1.

Detection of optical activities using a modulated circularly polarised beam is so sensitive that, as long as any optical delay is given at all to a minute spot beam by detect optical activities. What should be noted here is that as long as the piezo-optical modulator 10 incorporates an optically active crystal, the crystal axis should be taken into account. Namely, modulation efficiency will be higher if an incident polarised beam is adjusted according to the angle the beam forms with the axis of crystal. Thus, the polarised state of an incident beam is adjusted by means of a polarisation adjusting element 17 placed on the input side of the piezo-optical modulator 10. Needless to say, the polarisation adjusting element 17 may be so constituted as to allow an incident beam to pass through a half wavelength plate capable of rotating the beam, or to allow an incident beam to pass through a quarter wavelength plate to convert it into a circularly polarised beam, and then to permit a specific component thereof, say, a linearly polarised beam to exit therefrom. This example uses a piezo-optical modulator 10 incorporating an optically active crystal, but any other modulator can be used with the same advantage as long as it can give a periodic optical delay to an incident beam.

Placement of the mirror 4 in front of the analyser 5 is undesirable because the mirror may add an extra polarisation characteristic, and ideally the light path should not be bent. However, for a beam converged by the converging lens 4 to be guided to the light receiving element 7, it is necessary with a conventional transmission type scanning near field optical microscope to bend the light path by means of the mirror 4 for design convenience. The mirror incorporates a dichroic mirror instead of a conventional vapour-deposited aluminum mirror, thereby to lessen the difference in reflection of p- and s-polarised beams. Through this procedure it becomes possible to ignore the polarisation characteristic given by the mirror 4.

By virtue of an apparatus having the above constitution, a minute spot beam emanating from the minute opening interacts with the surface of sample 2, is converted, through that interaction, into a transmissive beam, passes through the sample 2 being given, during passage, optical activities including circular dichroism and optical rotation, and passes through the analyser 5 to be incident on the light receiving element 7 so that it may be registered there. This signal is rectified by the lock-in amplifier 8 which uses the reference signal (having a frequency of  $p$ ) delivered by the PEM driver 14 for rectification, and the rectified output is fed to the controller 16. When the  $2p$  component is submitted to lock-in rectification, it gives an optical rotation, and when the  $p$  component is submitted to lock-in rectification, it gives a circular dichroism. Thus, when these signals are submitted to the controller 16 to be converted into images in synchrony with



the scanning movement of the piezo actuator 15 as in a conventional scanning near field optical microscope, they will visualise the distribution of optical activities of the sample. Incidentally, if only the p component is required, the analyser 5 placed in front of the light receiving element 7 may be omitted.

Further, when not only the distribution of optical activities but also the absolute quantities of those activities are desired to be measured the ratio of the p component to the direct current component will give the ellipticity and the ratio of the 2p component to the direct current component will give the optical rotation. For the latter purpose it is not necessary to move the piezo actuator 15 so that the beam can scan over the sample along x- and y-axis directions, but to adjust the piezo actuator 15 such that a desired spot of sample 2 is placed close to the optical fibre probe 1 for measurement. By this process it is possible to quantitatively determine the optical activities of a very tiny area of a sample as with a conventional method based on the modulation of a circularly polarised beam.

Assuming that the optical fibre probe 1 gives an optical delay of  $\pi/2$  as a result of mechanical stresses as does a quarter wavelength plate, the electric field  $E_2$  of a beam as described by equation (2) above comes to be expressed by the following equation because of an optical delay given by the optical fibre probe 1.

$$E_x \propto \{1 + i \exp(i\delta)\} \quad (2')$$

As a result, the intensity I of light emanating from the analyser becomes:

$$I \propto \{R + (\Delta R/2) \cos \delta + R \sin (\Delta \theta + 2 \phi) \sin \delta\} \quad (5')$$

This equation, when resolved by a Bessel function, gives:

$$I \sim I(0) + I(p) \sin 2\pi p t + I(2p) \cos 4\pi p t + \dots \quad (6)$$

$$I(p) \propto \theta_k, I(2p) \propto \eta_k \quad (7')$$

What is worthy of notice here is that what the p and 2p components represent in this equation is opposite to what the same components expressed in equation (7) above represent.

The present invention can have the above-described constitution and operate in the above-described manner, and combines a scanning near field optical microscope with circular polarisation modulation thereby to make it possible not only to observe the distribution of optical activities of the sample 2 with a high sensitivity and resolution, but also to quantitatively determine the optical rotation and ellipticity of a specified tiny area of the sample. Further, as this method is based on a scanning near field optical microscope, it allows the production of a smaller apparatus with a lower cost than is possible with other similar observation means dependent on conventional techniques. The sample 2 may be in the atmosphere, in a liquid, or in a vacuum for measurement, and does not need to be thinly sectioned. Thus, this method does not impose any special restrictions on the preparation of the sample.

## (2) Example 2

Next, Example 2 according to this invention will be described with reference to Fig. 3 which shows the constitution of Example 2 of this invention. In Example 1 light is passed through a sample for measurement. In example 2 measurement is performed using light reflected from a sample. As the basic constitution and operation are the same as those of Example 1, parts achieving the same functions are represented by the same symbols, and their detailed explanation will be omitted.

The process the beam undergoes, after having emanated from the light source 11, till it exists from the minute opening of the optical fibre probe 1 as a minute spot beam are practically the same as that of the beam in Example 1. However, in example 2, a piezo-optical modulator 10 is not placed on a light path between the light source 11 and the optical fibre probe 1. As described above, the optical fibre probe 1 often has the property of retarding a beam. This is especially true when the optical fibre probe 1 is bent like a letter L so as to be operable in a dynamic AFM mode because the bent part causes a retardation in the beam. To avoid such inconvenience, a polarisation adjusting element 17 is placed on the input side of optical fibre probe 1 and the polarisation state of an incident beam is modified by that polarisation adjusting element 17 so as to adjust the polarisation characteristics of the minute spot beam emanating from the minute opening. The polarisation adjusting element 17 usually consists of a wavelength plate like a half-

wavelength plate or a quarter-wavelength plate, but the use of a compensatory plate will allow a more precise adjustment.

A minute spot beam reflected from the sample 2 after having interacted with the latter is converged by a converging lens 3. The converging lens 2 may be positioned at any place on the light path as long as it efficiently converges the minute spot beam reflected from the sample 2, or, instead of being made of a lens, it may be made of a light-converging mirror like a parabolic mirror, as long as it has a light converging activity. The beam, after being converged by the converging lens 3, is given an optical delay with a frequency of  $p$  (Hz) while it passes through a piezo-optical modulator 10, and then passes through an analyser 5 and filter 6 to be received by a light receiving element 7. As far as only the determination of circular dichroism of a beam from the  $p$  component is required, the use of analyser 5 may be omitted. As the remaining constitutions and operations are the same as those of Example 1, description of them will be omitted. Also with example 2 one can visualise the distribution of optical activities of sample 2 and quantitatively determine those optical activities with a high sensitivity and resolution.

Although in Example 1 the piezo-optical modulator 10 is placed on the input side of optical fibre probe 1, it is needless to say, the device in question may be put between the analyser 5 and sample 2 as in example 2. However, as the piezo-optical modulator 10 usually incorporates an optically active crystal, it is possible to efficiently give an optical delay to an incoming beam by directing the beam to the modulator 10 such that the polarisation plane of the beam has a specific angle with respect to the crystal axis. If the piezo-optical modulator 10 is positioned in such a way as to receive light reflected from the sample 2, the polarisation plane of the light will not take an optimum angle with respect to the crystal axis, and thus the modulation efficiency and detection sensitivity will often be worsened and lowered.

### (3) Example 3

Next, Example 3 according to this invention will be described with reference to Fig. 4 which illustrates the constitution of Example 3 of this invention. Although in Examples 1 and 2 an illumination mode is adopted whereby a beam emanating from a minute opening is allowed to illumine a sample, in example 3 a collection mode is adopted whereby a minute spot beam is detected through a minute opening. Further, a beam is allowed to pass through the sample 2. Parts having the same constitution or achieving the same functions as the corresponding parts of Example 1 are represented by the same symbols, and their explanation will be omitted.

A beam emanating from a light source 11 is given an optical delay during passage through a polarisation adjusting element 17 and piezo-optical modulator 10, and this is the same as in Example 1. The beam, after having been given an optical delay, is converged by a converging lens 19 into a convergent beam, and is incident through a side wall onto a triangular prism 21 carrying a sample 2 and converged to the bottom surface thereof. When the incident angle of the converged beam with respect to the bottom surface of the prism exceeds a critical angle, that beam is totally reflected by that bottom surface, and the side of the bottom surface facing the sample 2 gives rise to an evanescent beam. When an optical fibre probe having a minute opening at its tip is allowed to approach the sample 2 by the same method as used in Example 1, the evanescent beam present on the surface of sample 2, through interaction with the optical fibre probe 1, is converted into a transmissive beam which enters into the minute opening, transmits through the optical fibre probe 1, and exits from the other end of the same probe. The beam emanating from the other end of probe 1 is collimated by a collimator 20, and is allowed to pass through an analyser 5 and filter 6 to be received by a light receiving element 7. As the constitution of other elements and their operation are the same as those of Example 1, their explanation will be omitted. With example 3, it is also possible to obtain the distribution of optical activities of sample 2 or to quantitatively determine the optical activities thereof with a high sensitivity and resolution.

Example 3 uses the triangular prism 21 in such a way as to totally reflect an incident beam to produce an evanescent beam, but the total reflection may be produced by means of a dark-field illumination and, needless to say, the method is not limited to any specific one as long as an evanescent beam is produced on the surface of sample 2. It is also needless to say that, as in Example 1, the optical fibre probe 1 is allowed to illumine a tiny area of sample 2, and to receive a beam having undergone an interaction for measurement. In this case, the optical fibre probe must have, at the other end, a beam separating element like a beam splitter which separates a beam into an illumination component and a detection component.

In Examples 1, 2 and 3 described above, the optical fibre probe 1 has a minute opening at its tip, and radiates or transmits a minute spot beam through the minute opening for radiation or for measurement. It is needless to say, however the probe is not limited to any specific one. Preferably, it has a light transmitting body acting as a wave guide channel, being made of a comparatively transparent material to the wavelength of beams often used for measurement, like quartz or lithium niobate, and has a minute opening at its tip which is equal or less in size than the wavelength of the beam.

In Examples 1, 2 and 3 described above, the piezo-optical modulator 10 is installed, besides the optical fibre probe 1, to give an optical delay. However, many of the above-described light transmitting bodies can give an optical delay

through photoelasticity effects in the presence of an external force. Namely, instead of the piezo-optical modulator 10, a periodic force may be applied to a part of light transmitting body of the probe, and the resulting photo-elasticity effects may be utilised to give an optical delay changing in a periodic manner to a beam during the passage of the beam through the light transmitting body. Further, by adjusting the intensity of the external force, it is possible to alter the magnitude of the optical delay. As a result, it is possible to reduce the overall size of the apparatus.

Further, the method whereby the polarisation state of a minute spot beam is adjusted by a polarisation adjusting element 17 placed on the input side of a probe consisting of a light transmitting body is effective for the measurement combined not only with the modulation of a circularly polarised beam, but also with a cross-Nichols method. For example, although the optical fibre probe 1 may be bent in a form like a letter L so that the distance between the optical fibre probe 1 and sample 2 may be dynamically changed according to a dynamic AFM mode, the optical fibre probe 1 will then present an optical anisotropy which causes a retardation in a beam passing there through. As a result, even if a linearly polarised beam is fed to the optical fibre probe 1, only an elliptically polarised beam will emanate from the minute opening. This inconvenience can be avoided by inserting a polarisation adjusting element 17 in such a way as to cancel out the retardation, thereby allowing the minute spot beam to approximate to a linearly polarised beam. Therefore, when this arrangement is applied to a cross-Nichols method, the overall detection sensitivity will become higher than is possible with a similar apparatus dependent on the cross-Nichols method which uses an elliptically polarised beam emanating from the minute opening without any special treatment therefor.

In the above-described examples, the polarisation adjusting element 17 is installed, besides the optical fibre probe 1, to change the polarisation state of a beam before the beam is incident on the optical fibre probe 1. It is needless to say, however, that when a method is employed which consists of directly applying a force onto part of the optical fibre probe 1 and of controlling the intensity of that force, it is possible to alter the polarisation state of a beam during its passage through the optical fibre by way of photoelasticity effects resulting from the external force, thus allowing the optical fibre probe 1 also to act as a polarisation adjusting element 17.

A typical glass material has a photo-elasticity coefficient of about  $2-4 (10^{-6} \text{ mm}^2 \cdot \text{N}^{-1})$ , and stresses therein cause a retardation in a beam passing there through. Hence a beam passing through such a glass material undergoes a change in its polarised state. This can occur in the optical fibre probe 1 of Examples 1, 2 and 3 described above. When the optical fibre (particularly its core section) acting as a light transmitting body is exposed to a vibration externally applied, or vibrates itself while securely fixed, so that stresses develop within the fibre, a beam passing there through has its polarisation state altered as a result of photo-elasticity effects. The external vibration acts as a noise source to lower the S/N ratio, or only a slight shift of fixation may destroy the reproducibility of measurements. When the distance between the optical fibre probe 1 and sample 2 is controlled according to a dynamic AFM mode, the optical fibre probe 1 must be bent like a hook as with an AFM cantilever. To take such a form the fibre has to be bent while being heated, and the bent part has residual stresses. When a beam passes through this part, it receives an optical delay as a result of photo-elasticity effects. This not only applies to a part bent in the form of a hook, but also to the general form of an optical fibre probe 1 which is often not symmetrically configured with respect to the axis of light transmission, which results in the development of residual stresses.

To meet the above inconvenience, a probe is prepared whose light transmitting body is made of a material having a photo-elasticity coefficient of  $1 \times 10^{-6} (\text{mm}^2 \cdot \text{N}^{-1})$  or less. By preparing such a probe it is possible to ignore the effects of stresses caused by external vibrations and fixations, and to suppress the effects of residual stresses developing as a result of asymmetrical configuration with respect to the optical axis to a negligible level. For example, crown glass FK51 or FK52 provided by Shot Co. has a photo-elasticity coefficient of about  $0.7-1 \times 10^{-6} (\text{mm}^2 \cdot \text{N}^{-1})$ . Further, flint glass SF57 provided by Shot Co. which is composed of quartz containing a large amount of lead oxide has a photo-elasticity coefficient of about  $0.02 \times 10^{-6} (\text{mm}^2 \cdot \text{N}^{-1})$ . It can have a coefficient as small as  $0.005 \times 10^{-6} (\text{mm}^2 \cdot \text{N}^{-1})$ , depending on the ingredients contained therein. It is also possible to prepare an optical fibre probe from these glass materials. Thus, a light transmitting body made of a material whose photo-elasticity coefficient is  $1 \times 10^{-6} (\text{mm}^2 \cdot \text{N}^{-1})$  or less is utilised to form a probe, and with that probe it is possible to make a measurement where changes in polarisation characteristics due to external vibrations are effectively suppressed and a satisfactory S/N ratio is maintained. Further, even when the optical fibre probe 1 has its stem bent like a hook as in Examples 1, 2 and 3, it is possible to greatly reduce the changes in polarisation state which otherwise a beam would have suffered during passage through the bent part. As is evident from the above, preparing a probe from a light transmitting body made of a material having a smaller photo-elasticity coefficient is very useful for a measurement dependent on the use of a polarised beam including a cross-Nichols method as well as circular polarisation modulation.

Incidentally, without resorting to the preparation of a probe from a light transmitting body made of a material having a smaller photo-elasticity coefficient, it is possible to suppress the effects of residual stresses which may develop during the preparation of the probe by annealing the light transmitting body.

## (4) Example 4

In Examples 1 to 3, the light transmitting body consists of an optical fibre which has its tip sharpened, and has a minute opening at the probe tip. However, the method of illuminating a tiny area of a sample by a beam emanating from the tip of probe is not limited to radiation through a minute opening. There are a number of variants: an evanescent beam can be produced by plasmon deposited on the surface of a minute ball, or a grating whose lattice pitch is so shortened that the diffraction angle is lost. Further, the method whereby the tip of the probe can detect a beam emanating from a tiny area of the sample also has a number of variants: an evanescent beam may be produced on the surface of a sample by total reflection or by reflection from a surface-coated plasmon.

Besides the above a method may be employed whereby a cantilever, instead of a light transmitting body, which is produced after a semiconductor such as silicone or a metal has been sharpened, is utilised as a probe. Then, for example, the surface of the sample and the sharpened tip of the probe are illuminated by dark-field illumination as often used in microscopy; to cause a multiple scattering to occur between the sample and cantilever tip, and the scattered beam is measured by an external optical system.

Next, Example 4 of this invention wherein the probe does not consist of a light transmitting body will be described with reference to Fig. 5 which shows the constitution of Example 4 of this invention. A probe 22 may consist of a metal probe as used in a scanning tunnel microscope or a silicone cantilever as used in a scanning atomic force microscope, or other various materials. But, needless to say, it is not limited to any specific materials as long as the material has a high scattering efficiency. The sharpened tip of probe 22 is allowed to get close to a sample 2. The example depicted in the figure uses an optical lever to move the tip towards the sample, and as this tool is the same as those described earlier, explanation thereof will be omitted. The sample 2 is placed on a piezo actuator 15 and moves in x-, y- and z-axis directions.

A light flux emanating from a light source 11, after having passed through a polarisation adjusting element 17 and piezo-optical modulator 10, is converged by a collimator 20 and illuminates the sample 2 and the tip of the probe 22. This illuminating beam is given a periodically changing optical delay through the piezo-optical modulator 10. The light source 11 is usually a laser, and there is no element in the light path that may disturb the polarisation state of the beam passing along a light path such as the optical fibre probe as encountered in foregoing examples, but the polarisation adjusting element 17 is used to adjust the polarisation direction of an illuminating beam according to the condition of the sample 2.

As the tip of the probe 22 gets so close to the sample 22 that the distance therebetween is equal to or less than the wavelength of the illuminating beam, a multiple scattering takes place as a result of interaction between the surface of the sample 2 and the tip. This multiply scattered beam carries optical information regarding the surface condition of the sample 2 depending on the size of the tip of the probe 22. A converging lens converges this scattered beam which is then passed through an analyser 5 and filter 6 to be received by a light receiving element 7. As the subsequent processes are the same as those of foregoing examples, explanation thereof will be omitted. Adjusting the angles of light source 11 and of light receiving element 7 with respect to the sample 2, so that a beam emanating from the light source 11 to illumine the sample 2 and the probe 22 may not enter the light receiving element, will enable a selective pick-up of a multiply scattered beam, and achievement of a measurement with a satisfactory S/N ratio. As is evident from the above, even if a probe does not consist of a light transmitting body, it is possible to constitute a scanning near field optical microscope based on circular polarisation modulation, and to observe optical activities of a sample with a high sensitivity.

When a beam from the light source 11 radiates from one direction onto the sample 2 as in Fig. 5, entry of the radiating beam from the light source 11 into the light receiving element 7 should be avoided as much as possible. Further, this example is based on a transmission type apparatus, but what is mentioned above also applies to a reflection type apparatus.

Examples 1 to 4 described above use an optical lever to detect the displacement of the optical fibre probe 1 or the probe 22. However, the method is not limited to an optical lever or any other specific methods, as long as the method permits detection of the minute displacement of the optical fibre probe 1 or the probe 22. For example, when a dynamic AFM mode is employed, the probe may be applied on a quartz vibration detector, and the changes in vibration of the probe be followed by voltages delivered from the quartz vibration detector being monitored.

In Examples 1 to 4 described above, the method of controlling the distance between the optical fibre probe 1 or the probe 22 and the sample 2 is based on a dynamic AFM mode. However, the method is not limited to any specific modes, as long as the distance between the optical fibre probe 1 or the probe 22 and the sample 2 can be rendered to any small size at will: the distance in question may be adjusted according to a static AFM mode, or by light interference, or on the basis of a shearing force produced as a result of interaction between the two elements here concerned, or through the utilisation of a tunnel current. If the latter method is used, the optical fibre probe 1 or the probe 22 need not be bent like a letter L.

Although in Examples 1 to 4 described above, the distance between the optical fibre probe 1 or the probe 22 and

the sample 2 is measured and actively controlled on the basis of the measurement, the method is not limited to this. For example, the probe and sample may be allowed to move relative to each other to cause a current to flow therebetween, thereby maintaining constant the distance between the probe and sample surface by virtue of the viscosity of fluid as in a hard disk drive where the magnetic head floats by a certain definite amount (e.g., about 100nm) over the disk by virtue of an air bearing. Thus, the method does not necessarily require measurement of the distance between the probe and sample surface and a continuous monitoring of the proximity of the two elements here concerned. But, any method can be used with the same result as long as it allows the probe and sample surface to be close and constant to each other.

In Examples 1 to 4 described above, the light source 11 generates a laser beam composed of a single wavelength. However, a Xenon lamp may be used as the light source 11, and light therefrom may be used after it has passed through a spectroscope so that an appropriate component might be selected. In this case, measurement can be performed using beams with different wavelengths.

## Claims

### 1. A scanning near field optical microscope comprising:

a light source;  
a probe having a tip;  
means operatively to maintain the interval between the tip and a sample surface or medium within an action distance within which an interactive force results between the tip and the surface;  
means to obtain a polarised beam carrying optical information concerning the sample surface or medium, by allowing a beam from the light source to interact with the surface adjacent the tip;  
light receiving means to receive the polarised beam;  
modulating means placed in the light path between the light source and the light receiving means to establish a periodically changing optical delay in the beam.

### 2. A scanning near field optical microscope as claimed in claim 1, further comprising:

rectifying means to selectively separate, out of the output from the light receiving means, wave components having frequencies integer times as high as the modulation frequency of the modulating means.

### 3. A scanning near field optical microscope as claimed in Claim 1 or Claim 2 wherein the probe has a light transmitting body.

### 4. A scanning near field optical microscope as claimed in Claim 1 wherein the means to maintain the interval between the tip and the sample surface or medium within the action distance comprises:

moving means to alter the distance between the tip and the surface or medium;  
distance determining means to determine the distance between the tip and the surface of medium; and  
control means to maintain constant the distance between the tip and the surface or medium on the basis of output from the distance determining means.

### 5. A scanning near field optical microscope as claimed in Claim 4, wherein the distance determining means comprises:

vibrating means to vibrate the tip and the surface or medium relative to each other in a horizontal or vertical direction; and  
displacement detecting means to detect the displacement of the tip.

### 6. A scanning near field optical microscope as claimed in Claim 3, wherein the modulating means comprises means to impose a periodically changing stress in the light transmitting body.

### 7. A scanning near field optical microscope comprising:

a light source;  
a probe having a tip and a light transmitting body;  
means operatively to maintain the interval between the tip and the surface of a sample or medium within an action distance within which an inter-interactive force results between the tip and the surface or medium;

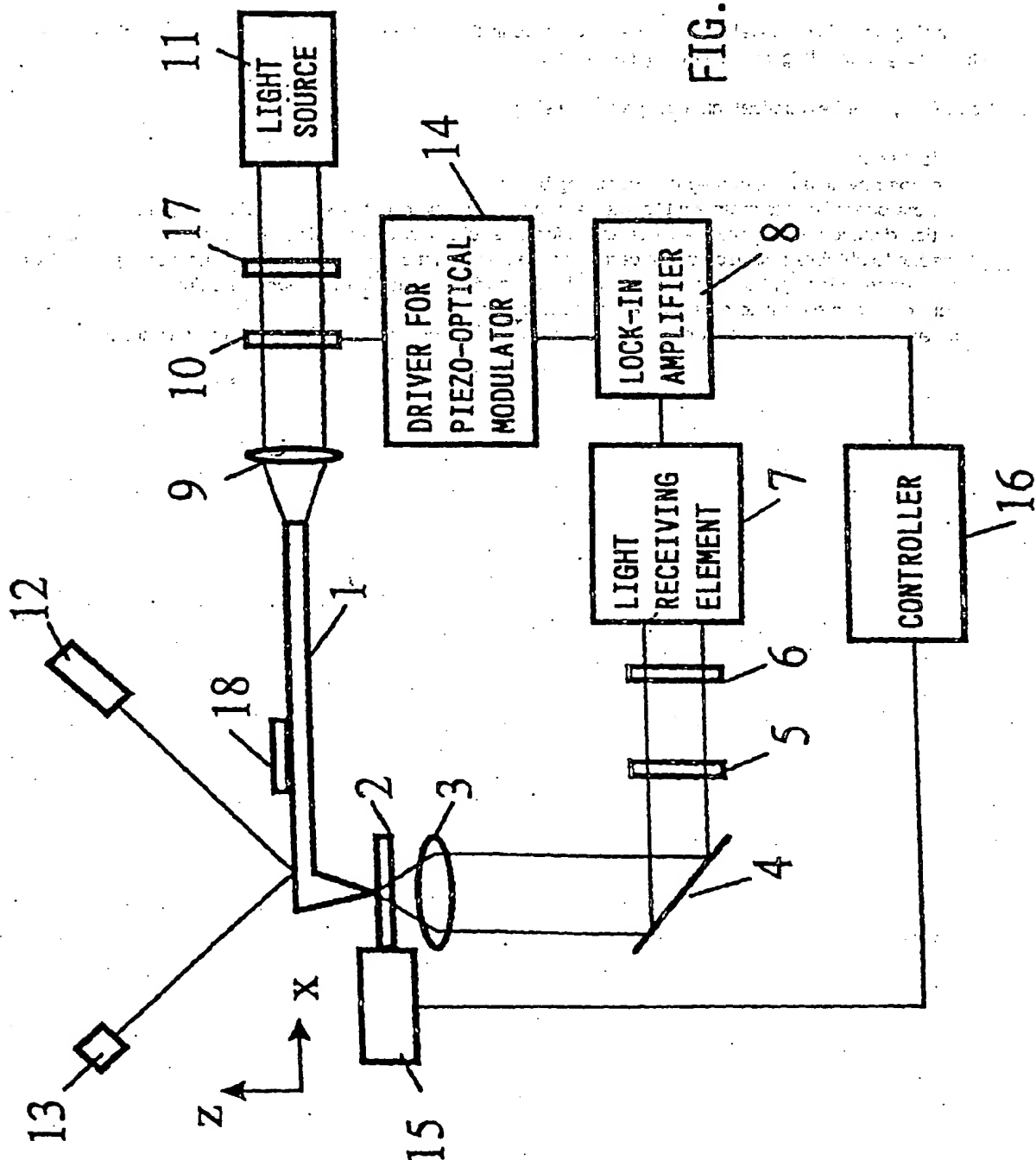
means to obtain a polarised beam carrying optical information concerning the sample or medium, by allowing a beam from the light source to interact with the surface or medium adjacent the tip; and light receiving means to receive the polarised beam, wherein a material constituting the light transmitting body has a photo-elasticity coefficient of  $1.0 \times 10^{-6}$  [mm<sup>2</sup>. N-1] or less.

8. A scanning near field optical microscope as claimed in Claim 7, wherein the material constituting the light transmitting body is quartz glass containing lead oxide.

9. A scanning near field optical microscope comprising:

a light source;  
a probe having a tip and a light transmitting body;  
means operatively to maintain the interval between the tip and the surface of a sample or medium within an action distance within which an interactive force results between the tip and the surface or medium;  
means to obtain a polarised beam carrying optical information concerning the sample or medium, by allowing a beam from the light source to interact with the surface or medium adjacent the tip;  
light receiving means to receive the polarised beam; and  
means to confer an optical delay by causing a stress to be generated in the light transmitting body.

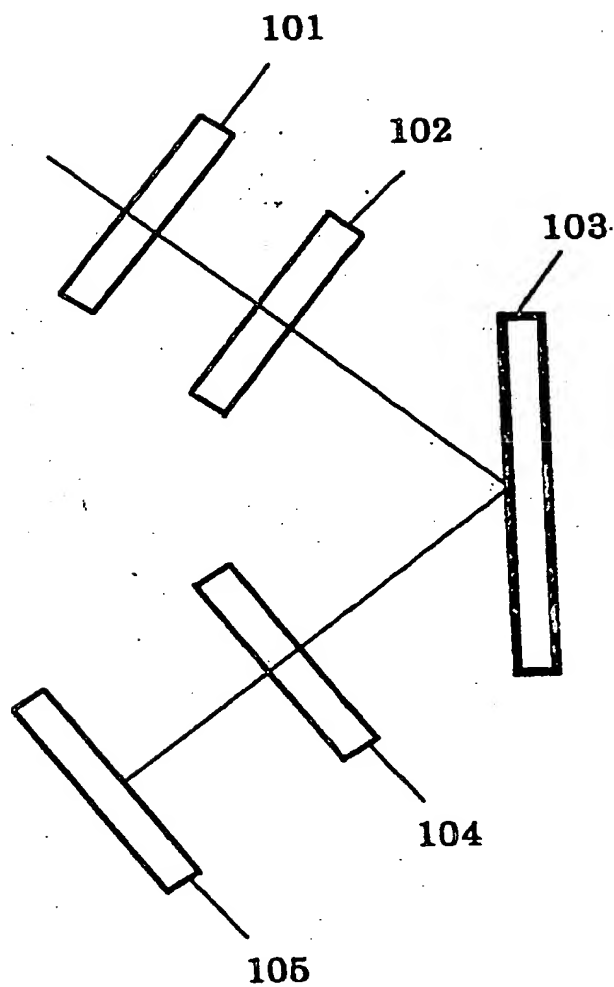
FIG. 1



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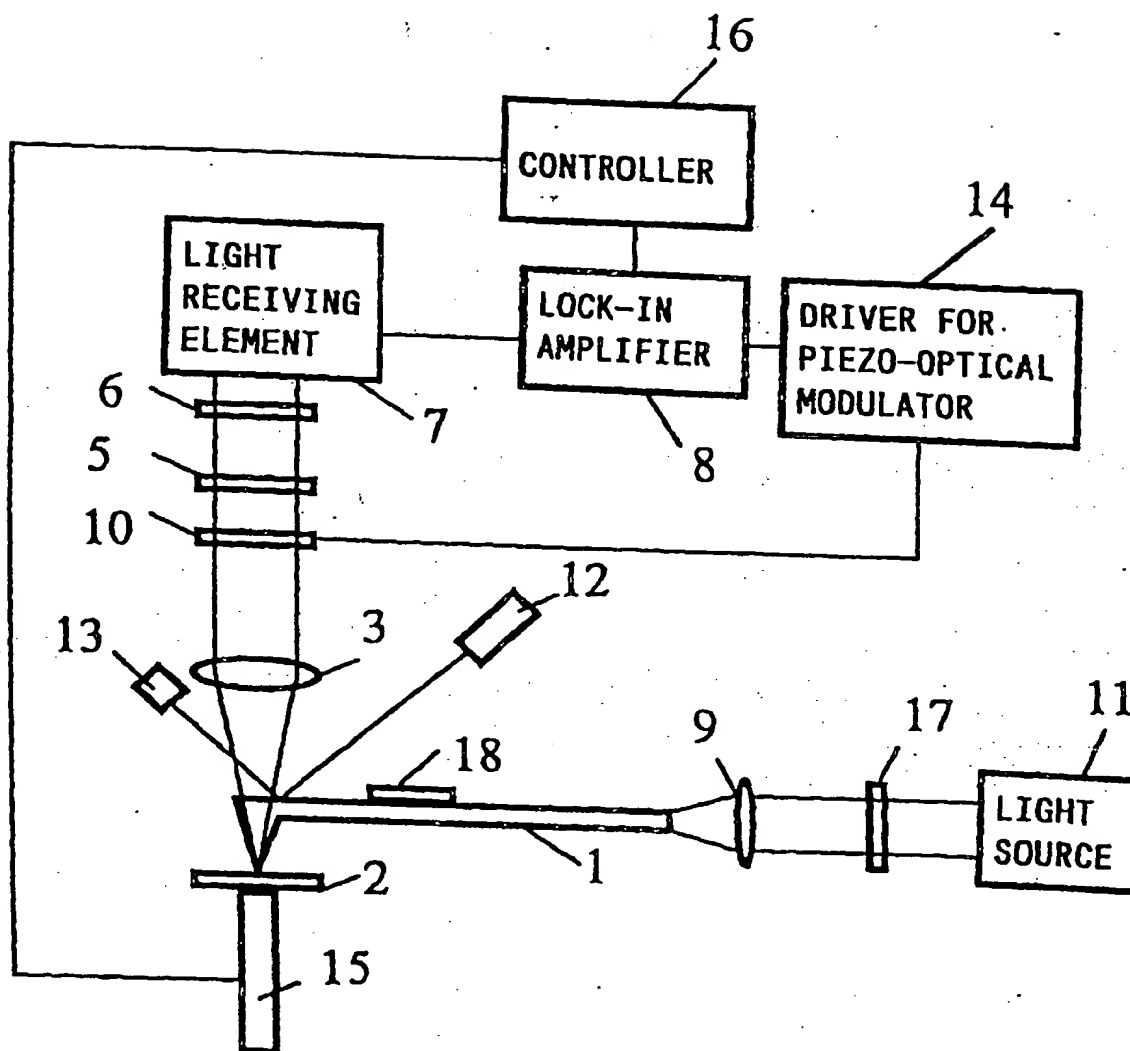


FIG. 2 PRIOR ART



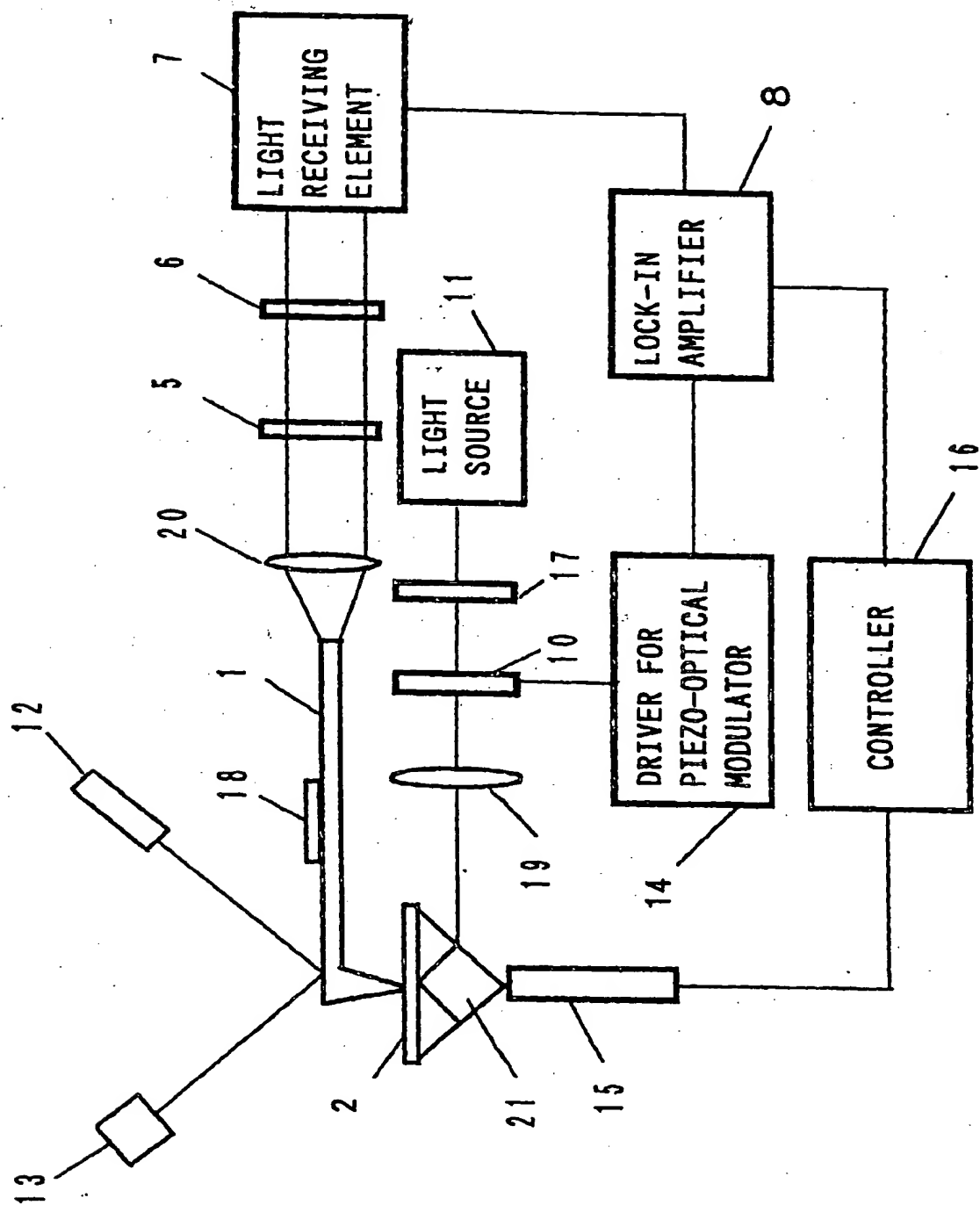
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FIG. 3



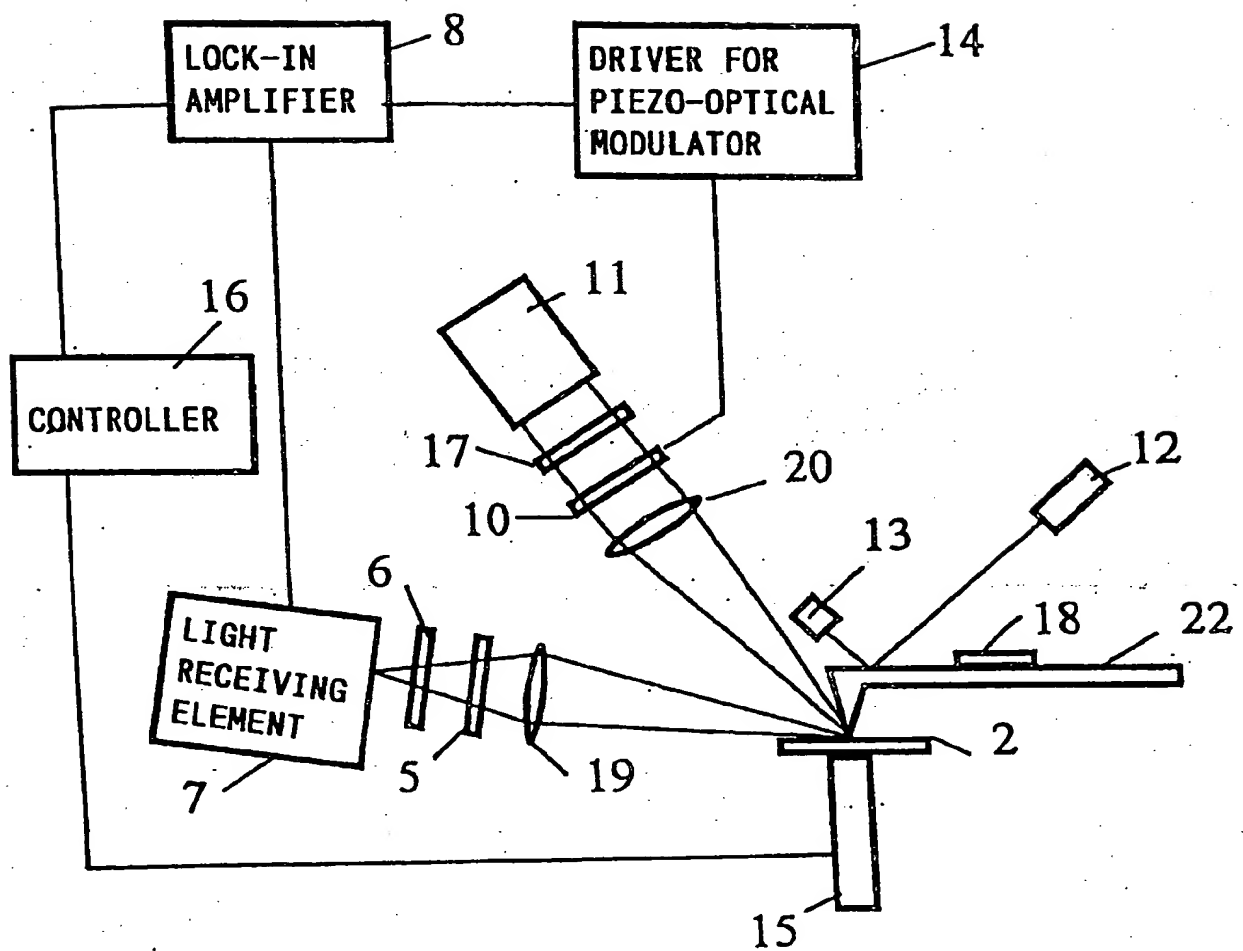
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FIG. 4



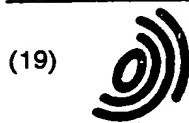
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FIG. 5



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### (54) Scanning near field optical microscope

(57) In a combined scanning near field optical microscope (Nsom) and atomic force microscope (AFM), an optical fibre probe (1) which has a minute opening on the top of a sharpened tip is brought close to a sample (2), and the probe is moved by a piezo actuator (15) along x- and y-axis directions so that a minute spot beam emanating from the minute opening can scan over the sample. For circular polarisation modulation to be incorporated in the process, a beam is given an optical delay, before it is incident on the optical fibre probe (1),

changing at a frequency of  $p$  (Hz) by means of a piezo-optical modulator (10). A minute spot beam emanating from the minute opening passes through the sample (2) to be received after passage through the sample (2) to be received after passage through an analyser (5) by a light receiving element (7). The output from the light receiving element (7) is fed to a lock-in amplifier, p- and 2p-components are separated through lock-in rectification, and they are rendered into images by a controller (16). It is used for measuring the distribution of magneto-optical effects.

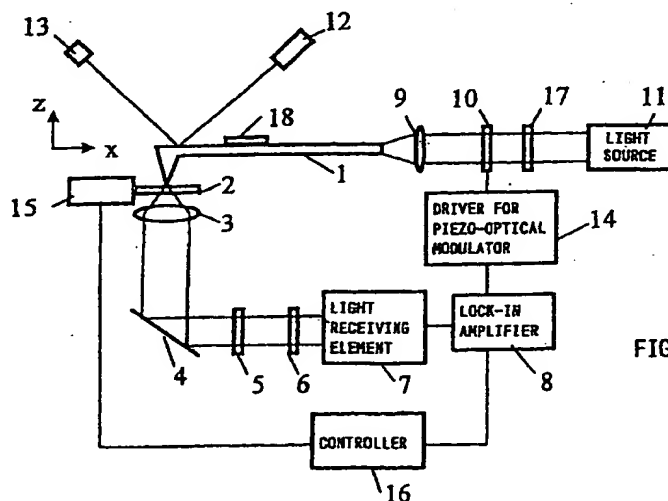


FIG. 1



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# EUROPEAN SEARCH REPORT

Application Number

EP 98 30 4109

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 6)
X	KOTTLER V ET AL: "Dichroic imaging of magnetic domains with a scanning near-field optical microscope" JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS, vol. 165, no. 1, January 1997, page 398-400 XP004058097	1,3-5	G02B21/00 G01B7/34 G01N21/21 G02B6/10
Y	* the whole document *	2	
Y	US 5 457 536 A (KORNFIELD JULIA A ET AL) 10 October 1995 * column 2, line 3 - line 19 * * column 5, line 58 - column 7, line 15 *	2	
X	WO 96 03641 A (KLEY VICTOR B) 8 February 1996 * page 20, line 10 - page 21, line 20; figure 1 * * page 51, line 24 - page 53, line 12; figure 11 *	1,3	
A	SILVA T J ET AL: "SCANNING NEAR-FIELD OPTICAL MICROSCOPE FOR THE IMAGING OF MAGNETIC DOMAINS IN OPTICALLY OPAQUE MATERIALS" APPLIED PHYSICS LETTERS, vol. 65, no. 6, 8 August 1994, pages 658-660, XP000464573 * the whole document *	1	TECHNICAL FIELDS SEARCHED (Int. Cl. 6) G02B G01B G01N G11B G02F
A	EP 0 674 200 A (SEIKO INSTR INC) 27 September 1995 * claim 1; figure 1 *	1,3-5	
-The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 11 August 1998	Examiner Ciarrocca, M
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background O: non-written disclosure P: intermediate document</p> <p>T: theory or principle underlying the invention E: earlier patent document, but published on, or after the filing date D: document cited in the application L: document cited for other reasons &amp;: member of the same patent family, corresponding document</p>			

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Application Number

EP 98 30 4109

### CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing more than ten claims.

- ☐ Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid, namely claim(s):
- ☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

### LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet B

- ☐ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- ☒ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:
- 1 - 5
- ☐ None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

European Patent  
Office**LACK OF UNITY OF INVENTION  
SHEET B**

Application Number

EP 98 30 4109

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

**1. Claims: 1-5**

Combined near field scanning optical/atomic force microscope using rectifying means to separate signals at integer harmonics of the modulation frequency.

**2. Claims: 6,9**

Combined near field scanning optical/atomic force microscope using stress in a light transmitting body to modulate the light beam.

**3. Claims: 7,8**

Combined near field scanning optical/atomic force microscope using a light transmitting probe tip with a photo-elasticity coefficient less than or equal to  $1.0 \times 10^{-6} \text{ m}^2/\text{N}$ .